April 13, 1965 C. C. WALLACE, JR 3,178,121

PROCESS FOR COMMINUTING GRIT IN PIGMENTS AND SUPERSONIC FLUID ENERGY MILL THEREFOR

Filed April 24, 1962

FIG. 1

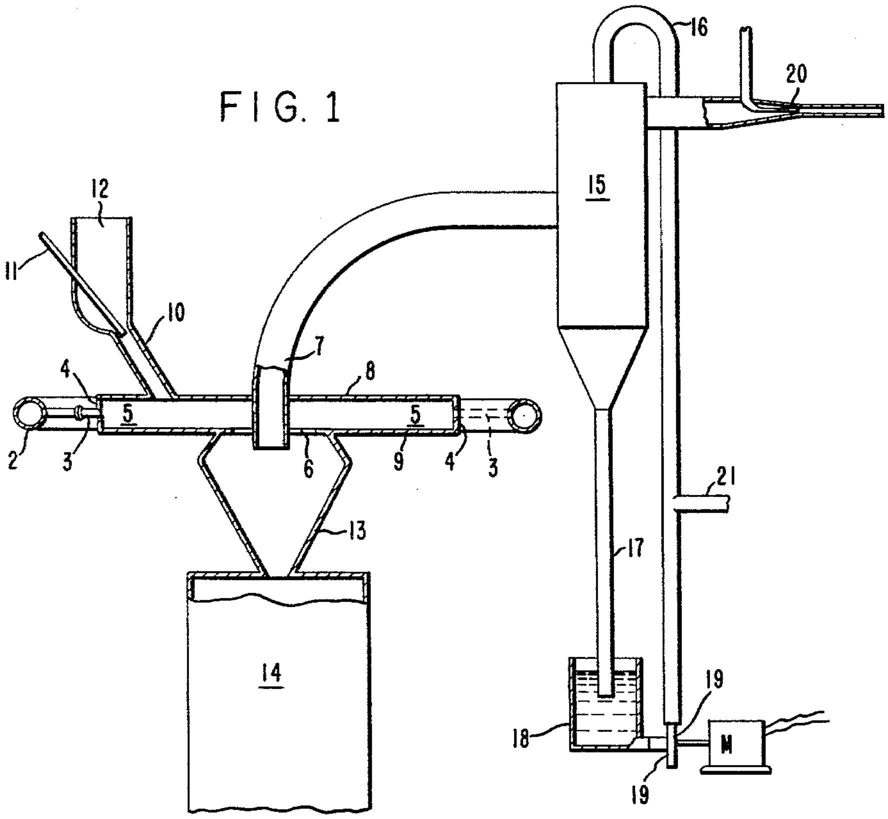


FIG. 2

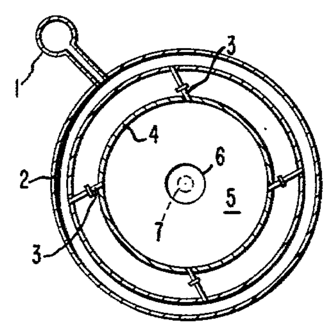
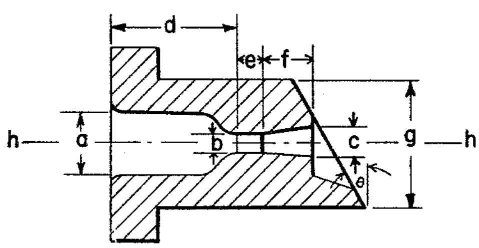


FIG. 3



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PROCESS FOR COMMINUTING GRIT IN PIGMENTS AND SUPERSONIC FLUID ENERGY MILL THEREFOR

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This invention relates to processes and apparatus for comminuting gritty, oversized particles in pigments, and is more particularly directed to the steps in such processes comprising (1) injecting a gaseous fluid tangentially and at supersonic velocity into the periphery of an enclosed circular space having an axial outlet, whereby to establish a disc-shaped vortex of said gaseous fluid inwardly spiraling to said outlet, (2) injecting grit-containing pigment into said vortex at a locus remote from said outlet, whereby the grit is comminuted in the vortex, and (3) discharging the gaseous fluid, pigment, and comminuted grit through the outlet while maintaining the pressure therein at 2 to 30 p.s.i.a.; is further particularly directed to apparatus adapted for carrying out said processes, the apparatus comprising, in combination, a circular, disc-shaped chamber having a peripheral wall through which said chamber is in closed communication with a source of pressurized gaseous fluid through a multiplicity of converging-diverging nozzles, each nozzle having inlet bore, throat bore, and exit bore portions, all of circular cross section and having a common axis, the inlet bore converging through a neck to a cylindrical throat having a diameter about one-third that of the inlet bore, said throat opening into a tapered outlet bore of diameter increasing linearly in the direction of a discharge outlet in said exit portion, the taper angle between the wall and axis of the outlet bore being about 2 to 14°, said exit portion having a truncated exit face, the angle between the truncating plane forming the exit face and a plane perpendicular to the axis of the outlet bore being about from 19 to 42°, and there being in the truncated face an inset face around the discharge opening of the outlet bore, perpendicular to the axis thereof and extending out from said discharge opening a distance of from ½ to 3 radii of said opening, the opening defined by said inset face tapering outwardly from the periphery of said face to the truncated face at an angle of at least about 6° from the axis of the outlet bore, said nozzles being mounted in said peripheral wall at substantially equally spaced points therein, with the truncated face of each nozzle substantially flush with said wall and the axis of the nozzle bore being tangent to a circle, concentric with said chamber, of a radius from ⅓ to ⅔ the radius of said chamber, the chamber having an axial discharge port concentric with its periphery, means communicating with said chamber for feeding pulverulent solids into it between its periphery and the discharge port, and means communicating with the discharge port for separating effluent solids from gaseous fluids; and is still further directed to the converging-diverging nozzles used in said apparatus.

In the drawings:

FIG. 1 is a sketch, not to scale, showing in vertical cross section an apparatus of the invention, and

FIG. 2 is a horizontal cross section, also not to scale, illustrating in more detail the vortex chamber and accessories for supplying steam thereto, and

FIG. 3 is a cross section drawing, to scale, of a nozzle of the invention, said nozzle being adapted for injecting steam at supersonic velocity into the vortex.

It is common practice, in the production of inorganic pigments such as titanium dioxide, to subject the pigment to a final grinding operation. Dry grinding is frequently used to supplement wet grinding because it gives improvements in certain pigment characteristics such as oil absorption and dispersibility. An additional object of dry grinding is to improve the fineness of the pigment in the sense that coarse particles, usually referred to as "grit," are reduced in size. Many grinding operations are followed by a size classification step which removes residual coarse particles. In preparing a white pigment, such as titanium dioxide for use in enamel paint, it is essential to minimize the amount of grit as much as possible since any grit is readily noticed as an impairment in the gloss of the paint surface. It is therefore an object of the present invention to provide improved methods and apparatus for dry grinding pigments to minimize the amount of grit therein.

The titanium dioxide pigment may be prepared by well known procedures involving hydrolytic precipitation from a sulfate solution of ilmenite followed by filtration, washing and calcination. The calcination at temperatures up to 1000° C. is required to develop the proper size of fine crystallites of high refractive index. Although these crystallites are less than one micron in diameter, they become cemented into gritty agglomerates which must be disintegrated. Another method of making TiO_2 pigment involves the high temperature oxidation of $TiCl_4$. This product also contains oversized agglomerates which must be comminuted.

One of the most effective devices for breaking these agglomerates into pigmentary particles is known as a "Micronizer." A description of this type of equipment is given in Chemical and Engineering Progress vol. 55, January 1959, pp. 108–10 and also in U.S. Patent 2,032,827. The Micronizer is one of a family of fluid energy mills. It differs from the other types of fluid energy mills in that it has a circular disc-shaped working chamber in which both grinding and size-classifying steps are carried out simultaneously.

The grinding of white pigment to the degree of fineness necessary to yield highest gloss in an automotive finish, or in the pure white refrigerator enamels, is hampered by the undesirable side effects encountered in most of the commercial methods. It is always a problem to maintain the intrinsic whiteness of the pigment because the wear and errosion of the grinding equipment causes contamination. The fluid energy mills are probably the least damaging of the known mills in this respect, but it has become necessary to construct the parts, which come into violent contact with the pigment, of very hard materials such as Haynes "Stellite." It is therefore a further object of this invention to provide improved methods and apparatus for minimizing grit in pigments without degradation of the color of the pigments.

Now according to the present invention it has been found that the velocity at which the gaseous fluid is injected into the vortex of a fluid energy mill of the "Micronizer" type has a hitherto unrecognized bearing on the completeness of comminution which can be achieved therein, and particularly, that if this velocity is supersonic, the degree of completeness obtainable is higher than previously considered possible. The arrangement of means whereby comminution of solids by such supersonic injection is accomplished, and the nozzles of novel design whereby supersonic gas injection velocities are achieved are further unobvious aspects of the invention. By application of the novel processes and apparatus, grit in pulverulent pigments such as titanium dioxide can be reduced to an amount too small to measure, without degradation of other essential pigment qualities such as whiteness.

In the description which follows the invention will be described with especial reference to superheated steam, but it will be understood that the gaseous fluid can be another medium, such as air. It will further be understood that the pulverulent solid, such as grit-containing pigment, can be fed into the vortex by conventional means such as mechanical feeders or by injection with steam or air. In the latter case, unless otherwise specified, the amounts of gaseous fluid used for injecting the pulverulent solid are not included when stipulating such factors as gas flow rates.

The gloss improvement achieved in pigments comminuted according to this invention is best obtained under conditions which break down the larger particles and do not produce appreciable quantities of superfine particles. This is the reason for selecting the disc-shaped grinding zone, since the violent vortex action obtained at the steam velocities used effects a separation of the particles according to size. The coarser particles are thrown by centrifugal action to the periphery where the attrition is greatest, while the fines are quickly removed to product collection without being over-ground. The actual grinding action takes place near the periphery of the vortex. Under ordinary conditions the pigment suspension whirls about this periphery undergoing attrition and size reduction as the particles collide with each other both as they flow along near the peripheral wall and in the turbulent spots where the circular flow intersects the incoming jets. In such a grinding zone the amount of kinetic energy imparted to the pigment particle will determine the vigor of its impact with particles traveling in another direction and consequently the amount of fracture and size reduction obtained.

According to this invention the velocity of the steam entering the vortex through the peripheral jets is increased above the sonic velocity for the steam. This introduces a very new and different set of conditions in the peripheral grinding zone. In the first place, the kinetic energy transferred to the particles may be an order of magnitude higher than previously possible. Then, with the nozzle exit velocity in excess of sonic velocity a new element is introduced into the grinding zone, namely a shock wave which is formed when the supersonic steam leaves the nozzle and expands freely, i.e. non-isentropically, into the outer vortex. This expansion causes the velocity to drop quickly to sonic velocity just inside the periphery, generating a standing wave in the vicinity of which tremendous turbulence exists. The whirling pigment collides with the wave and the turbulent zone where it acquires great kinetic energy and undergoes very effective particle-to-particle collision because of the extreme turbulence. It is believed, too, that the supersonic jets tend to keep the major grinding action away from the apparatus walls, reducing the wear and yielding a whiter product.

An essential feature of this invention lies in increasing the kinetic energy of the pigment particles or aggregates by imparting supersonic velocity to the steam which enters the particle suspension. This is accomplished by means of nozzles which are designed for substantially isentropic flow. They are of the converging-diverging type. The bore of the nozzle has an initially decreasing diameter in the direction of the steam flow to a minimum value at a throat section when it begins to increase or diverge. By maintaining sufficient pressure drop across such nozzles supersonic velocities are attained at the exit. The design of converging-diverging nozzles is discussed in Shapiro's "The Dynamics and Thermodynamics of Compressible Fluid Flow" referred to below. The supersonic stream leaving these nozzles, as used in this invention, enters the swirling vortex at an angle which augments the rotary motion. It intersects and mingles with the paths of the swirling particles transferring high kinetic energy to them to give the improved size reduction.

In a preferred process of the invention the vortex is established and maintained by jetting superheated steam at supersonic velocity through a multiplicity of nozzles of novel design tangentially mounted around the periphery of the enclosed space. The steam is fed to these nozzles at a pressure ranging upward from 100, preferably 140 to 600 p.s.i.a., temperatures in the 400 to 800° F. range, and in a superheated condition. Preferably at least 30° F. of superheat is provided at this point. The discharge zone pressure is maintained in the range of from 2 to 30 p.s.i.a. to obtain a practical pressure drop across the vortex. This pressure drop, the total cross section of the nozzle bores, and the design of the nozzles are correlated with the quantity of steam used, and the inlet pressure and temperature are adjusted to impart supersonic velocity to the steam in the exit portion of the nozzles. This velocity may be as great as Mach 10, but it is preferably set at values of from Mach 1.5 to 3. The ratio of nozzle steam to the pigment fed, measured in pounds per hour, is preferably in the range of from 1 to 5. The total flow of steam through the nozzles is maintained at at least 15 pounds per hour per square inch of vortex rim. The fine, comminuted pigment is carried by the steam out through the discharge port and recovered by known means, including wet and dry methods.

In an alternative procedure, at least a portion of the pigment may be introduced into the disc-shaped vortex zone along with the steam flowing through the nozzles. This pigment may pass through the full length of the nozzle but this engenders problems of wear and choking at the throat. Preferably, the pigment is introduced by suitable means such as screw feeders etc. into the diverging end of the nozzle where supersonic flow is already established. The particles then undergo very rapid acceleration and gain high kinetic energy for the impact against other particles swirling at lower speeds in the vortex. In most cases the nozzles used are quite small in inside diameter so that only a portion of the total pigment is fed through them. The nozzles may be redesigned to take into account the volume of pigment being fed.

An important factor to be considered in practicing this invention is the weight ratio of nozzle steam to pigment feed. If the relative rate of steam flow is too high the steam costs become prohibitive. On the other hand too low a ratio of nozzle steam to pigment, which might be due to nozzles which are too small or too few, or too rapid a rate of pigment feed, results in choking of the chamber, reduction in vortex action, and inferior classifying action in the inner vortex. For this reason the weight ratio of nozzle steam to titanium dioxide pigment ranges from 0.5:1 to 3:1. When part of the total steam is used for injecting the pigment, one uses as much as necessary for proper feeding of the pigment, and uses the rest through the supersonic nozzles.

The total steam throughput can be considerably varied. A certain minimum steam flow is necessary, for a given size of grinding chamber, to maintain a sufficient vortex speed. The vigor of the grinding action is greater with higher peripheral velocity, while complete classification of the oversize particles and the return of them to the peripheral grinding zone requires quite high velocity in the mid-portion of the vortex. This dual action is best optimized by relating the flow of nozzle steam to the area of the periphery, or rim, of the vortex. For instance, in titanium dioxide practice, when the chamber diameter is 36" and its height 1½", the peripheral area is 170 sq. inches, and the nozzle steam is fed at 4500 #/hr. or 26.5 #/hr./sq. in. of vortex rim. There appears to be no theoretical upper limit to the amount of steam used, but in practice very high flows become uneconomical because of the high nozzle entrance pressure required. Preferred limits are between 18 and 30 pounds of steam per hour per sq. in. of peripheral area.

Having provided for the supersonic flow and resulting high kinetic energy in the system, the relative rates of feed (steam and pigment) as well as the amount of steam for a given size of chamber are established as previously discussed to get the full improvement described.

Novel apparatus especially adapted for carrying out the processes of this invention is illustrated in FIGURE 1 and FIGURE 2 of the drawings. Reference characters are the same for identical parts in the two figures. FIGURE 3 shows details of the supersonic nozzles used in FIGURES 1 and 2.

In FIG. 2, 1 is a source of superheated steam having temperature- and pressure-controlling capabilities and 2 is a steam header encircling the peripheral wall 4 of circular grinding chamber 5. The nozzles 3, of which only four are shown, interconnect the steam header and the grinding chamber. The wall of the cylindrical discharge port 6 and the steam exhaust duct 7 are axially located. Each nozzle, 3, enters the chamber wall 4 at an angle such that the extension of the nozzle axis is tangent to a circle about the center of the chamber which as a radius not more than about ⅔ nor less than ⅓ the radius of the chamber. A multiplicity of these nozzles is used, sixteen being about right for a chamber of 36 inches diameter.

In FIG. 1 the whole assembly is essentially shown. The chamber 5 is shown to be rather flat and disc-shaped, being determined by the upper and lower plates 8 and 9 and the peripheral wall or rim 4. The nozzles 3 enter chamber 5 at an angle to the plane of the drawing. 10, 11, and 12 illustrate a feeding device for introducing the material to be ground. The steam jet 11 operating in the venturi section of inlet 10 draws the powdered feed from the hopper 12 forcing it into the chamber. This inlet 10 is set at an angle to the plane of the chamber 5 and usually somewhat tangentially, to facilitate flow of the solids and steam into the chamber vortex. Although it does not enter chamber 5 at the periphery it is nevertheless considered to enter at a locus remote from the chamber outlet because powder fed therethrough enters the vortex rather than the discharge zone. The cylindrical discharge opening formed by discharge port 6, in conjunction with the conical enclosure 13, forms a centrifugal separator into which the ground product settles while the steam flows out through exhaust duct 7 to the condenser 15, which is fed by cold water from pipe 16. A barometric leg 17, with its lower, open end submerged in water in sump 18 receives the condensed steam containing small amounts of fine product. A steam eductor 20 connected to the condenser 15 is adapted to lower the pressure therein and hence in duct 1 and discharge zone 6. The water in sump 18 is recirculated by pump 19 through line 16 to operate the jet condenser. Condensate containing finely divided product is drawn off through bleed-line 21 to product recovery. The product separating in 13 settles into an insulated storage silo 14 which is kept hot enough to prevent condensation of the steam. This silo 14 is kept substantially closed to prevent inleakage of air.

The design of a supersonic nozzle suitable for use as nozzle 3 of FIGURES 1 and 2 is shown in detail in FIGURE 3. Thus, FIGURE 3 is a cross section of such a nozzle having, from left to right, inlet bore, throat bore, and exit bore portions, all of circular cross section and having a common axis, h. The inlet bore, total length d and diameter a, converges through a neck to a cylindrical throat having diameter b about one-third that of the inlet bore, and length (e). The throat opens into a tapered outlet bore, the diameter of which is the same as that of the throat at the end adjacent the throat but increases linearly in the direction of a discharge outlet of diameter c in the exit portion, the length of the outlet bore being shown as f, and the taper angle between the wall and axis h of the outlet bore being about 6°. The exit portion has a truncated face, the angle θ between the truncating plane forming the exit face and a plane perpendicular to the axis h being about 30°. In the truncated face there is an inset face around the discharge opening of the outlet bore, perpendicular to axis h, and about twice the diameter of the discharge opening, the opening defined by said inset face tapering outwardly from the periphery of the face to the truncated face at an angle of about 15° from axis h. In FIGURE 3 the outside diameter of the nozzle is g.

In the specific design of FIGURE 3 the nozzle has diametral dimensions a (inlet)=0.5″, b (throat)=.156″, and c (outlet)=0.239″ (which latter two dimensions define an expanding divergence in the nozzle outlet of 6° radially), and g (outside diameter)=0.997″. The nozzle has length dimensions of d (converging inlet)=1.020″, e (throat length)=0.156″, and f (diverging outlet)=0.395″. The transition from the inlet bore to the nozzle throat is accomplished through a neck defined by reversed ¼″ radii drawn over lengths of ⅛″ each.

The design of the converging-diverging nozzles can be varied considerably from the specific embodiment shown in FIGURE 3. The dimensions given, for instance, are illustrative and are not to be construed as limiting except as otherwise indicated. The taper angle between the wall and axis of the outlet bore can be about from 2 to 14°. The angle between the truncating plane forming the exit face and a plane perpendicular to the axis of the outlet bore can be about from 19 to 42°. The inset face around the discharge opening of the outlet bore need not be circular, but rather can be oval or eliptical, provided that the opening it defines is of acceptable aerodynamic character in that it does not cause damaging turbulence to be generated in use. This face can extend out from the discharge opening of the outlet bore a distance of from ½ to 3 radii of said opening. The outward taper from the periphery of the inset face to the truncated face is at least about 6° from the axis of the outlet bore.

The manner of mounting the nozzles in the peripheral wall of the circular chamber is very important. They should be placed at substantially equally spaced points around the periphery. The axis of the bore of each nozzle should be tangent to a circle, concentric with the chamber, of a radius from ⅓ to ⅔ the radius of the chamber. The truncated face of the nozzle should be flush with the inner wall of the chamber, and any protuberance, or recessing other than that formed by the inset face, is strictly to be avoided. In practice it is advantageous to provide a keyed portion on the nozzle or some other indexing device to insure perfect alignment. While the truncated face is described herein as defined by a plane, it will be understood that this plane may be curved slightly to conform perfectly to the curvature of the peripheral wall without departing from the scope of the invention.

The design of the inset face of the nozzle and the space defined thereby provides a maximum of assistance to the gaseous fluid entering the vortex at supersonic velocity. On the other hand it provides a structure in which erosion of nozzles and chamber walls is minimized. Thus there is a novel and unobvious cooperation between the nozzle design and the comminuting apparatus, particularly since the overall result is improved comminution in the vortex.

The apparatus of this invention embodies several features in combination to produce effects not attainable in other fluid energy mills. The circular grinding chamber in which a violent vortex is formed, in combination with the supersonic tangential nozzles, subject the larger particles of the material being ground to repeated high energy impacts in the region of the shock waves caused by the incoming supersonic jets of gas while the material is simultaneously sized by the vortex action to separate the fine product. This prevents overgrinding of fines as well as increasing the efficiency and capacity of the mill. The fact that these operations occur simultaneously in one vortex while high kinetic energy levels are maintained is believed to be the main reason that the apparatus of this invention achieves more thorough elimination of the coarse fraction or "grit" while operating at high throughput and efficiency. In other mills, such as fluid energy tube mills, the material passes through the grinding zone and then on to a separate classifying zone where kinetic energy is lost and must be supplied again to the coarse fraction. Consequently the average kinetic energy of the particles to be comminuted attains a much higher level in the machine of this invention than in other fluid energy mills.

In the apparatus of this invention the nozzles can be so designed and arranged as to give jet velocities as high as Mach 10, with Mach numbers of 1.5 to 3 being easily attained with practical steam pressures. Since the kinetic energy of the moving particles increases with the square of the velocity it is easily seen that the grinding rate due to impact fracture is greatly increased over that from impacts due to velocities below sonic.

To further improve the action of this mill a pressure-lowering device such as the jet condenser **15** of FIG. 1, can be connected with the discharge zone of the vortex chamber. By lowering the pressure below atmospheric pressure two improvements are obtained. First, the lower pressure desirably creates a greater radial pressure drop across the chamber and also across the nozzles which in turn increases the jet velocity. Such a procedure, when applied to conventional mills having straight bore or merely converging nozzles, will not increase the jet velocity to more than sonic velocity. Secondly, the lowered pressure in the discharge and product collecting zones prevents condensation of water and consequent fouling or degradation of the product. Whether or not pressure-lowering devices are used, care should be exercised to provide an adequate discharge opening at the center of the vortex, as shown at **6** in FIGS. 1 and 2, so as to avoid pressure build-up.

Certain modifications and variations in the arrangement of the apparatus may be made as long as the essential features required to give the improved results are maintained. The source of steam and the steam header may be located above or below the chamber, it being only necessary that steam be supplied to the nozzle at the desired temperature and pressure. The use of the supersonic nozzles tangentially placed as described is essential. It is also essential that the chamber be circular and disc-shaped. The flat cover plates as shown in the drawing may be varied somewhat; for example they may be sloped to make the center of the chamber thicker.

The method of separating the comminuted product from the steam and collecting it may vary. For example, the whole product may be conducted to a condenser where the steam is condensed and a slurry formed. On the other hand, dry recovery from the stream can be accomplished by means of filters.

In a preferred adaptation the added feature of means for reducing the pressure at the center or discharge zone of the chamber comes within the scope of the invention. This means may comprise the jet device shown or may be a pump, fan or turbine which will withdraw the steam to give the desired lower pressure.

Various materials of construction may be used. Ordinary steel is usually satisfactory although the points of extreme wear which include the nozzles themselves and the rim wall of the chamber are preferably made of hardened material such as Haynes "Stellite" or a surface-hardened metal.

The operation of the device has here been described in terms of using superheated steam as the vaporous or gaseous fluid medium. This is done because steam can be supplied at these pressures quite economically, also because the pigment is easier to disperse in steam. The ability of steam to be condensed to liquid is often advantageous. However, it is to be understood that any other gas or vapor, substantially innocuous toward the solids being ground, may be used.

Examples

The invention will be better understood by reference to the following illustrative examples.

Example 1

This example illustrates the applicability of the invention to improvement of the quality of a sulfate-process type titanium dioxide pigment.

A commercial "micronizer" was modified to provide the desired conditions. This machine had a horizontal working chamber 36" in diameter and 1½" deep in the shape of a flat disc defined by upper and lower flat steel plates and a circular peripheral wall, which was made of "Stellite" to resist wear. A discharge port in the center of the bottom plate was connected in series with a cyclone separator, a condenser and a steam eductor. The eductor served to lower the pressure at the discharge port to about 5 p.s.i.a. Sixteen steam nozzles were mounted substantially equidistant in the "Stellite" wall in the center plane of the working chamber and at a 30° angle with the tangent to the peripheral wall. These nozzles were of the novel design illustrated in FIG. 3 of the drawings and replaced those supplied with the machine. They were of the converging-diverging type with inlet dia.=0.5 in., throat dia.=0.156 in., and outlet diameter of 0.239 in. A toroidal steam header encircled these nozzles and supplied steam to them at 290 p.s.i.a. and 460° F. With this steam pressure and temperature it was calculated that the steam velocity in the nozzle exit would be Mach No. 1.96. For calculation of Mach numbers see "Rocket Propulsion Elements" by G. P. Sutton, John Wiley, 1956; also, A. H. Shapiro, "The Dynamics and Thermodynamics of Compressible Fluid Flow," vol. 1, the Ronald Press, New York, N. Y. A feed port was located in the top plate 15" from the center. It comprised the outlet of a tube of 2.4" I.D. and was set tangentially at a 20° slope, through which the pigment was fed by steam injection at a controlled rate from a hopper through a star valve acting as a seal against air. A sulfate process, calcined TiO_2 pigment in granular form was used in this run.

To start the process, the superheated steam was fed via the header through the nozzles for several minutes until the apparatus reached thermal equilibrium and the initial condensate in the working chamber and cyclone separator evaporated. The pigment feed and injector steam were then started and the following flows established.

Nozzle steam
(total) ---- 4500#/hr. (26.5#/hr./sq. in. vortex rim).
Pigment ----- 2500#/hr.
Injector
steam ----- 1100#/hr.

The conditions of temperature and pressure mentioned above were maintained and the run continued for 24 hours. The product pigment was sampled each hour and composited for quality tests. As a control for this test run a second "micronizer," of the same size and design characteristics except for the special nozzles, was similarly operated using the same pigment feed and the same steam and pigment temperatures. The standard nozzles were of the straight-bore commercial design with a 17/64 inch inside diameter. Quality tests on the product from the two micronizers were as follows:

	Experimental run	Control
Color	18:3y	17:4y
Carbon Black Undertone	9	8½
Gloss	61	53

The above quality tests are described as follows:

Color.—This is a method of visually evaluating the relative brightness of white pigments. The samples are graded by being placed side by side with a standard pigment of an arbitrarily assigned brightness number. A series of these standards is set up according to a scale of brightness numbers, the higher the number the brighter the standard. A difference of one point is about the least discernable difference to a trained eye. In this case the final standard used for comparison was rated at 18:2*y*. The "*y*" values indicate degrees of yellowness which is very slight yellow cast usually found in rutile TiO_2 pigments. A description of this test by J. E. Booge and H. E. Eastlack is found in the Paint, Oil and Chemical Review, April 9, 1924.

Carbon black undertone.—This test relates to the fineness and is described in Schaumann U.S. Patent No. 2,502,247. The test is carried out by mulling an oil paste of the pigment to be tested with a standard amount of a carbon black and comparing this paste, in a side-by-side draw-down, with a paste prepared in the same way with a standard pigment. The 0 to 100 numerical scale is set up on which the standard pigments are rated. The higher numbers indicate finer particle size and a generally more desirable pigment from the point of view of use in gloss enamels and for use as a base for blending with colored pigments.

Gloss.—"Gloss" is a term used to describe the optical smoothness of a reflecting surface such as that of an enamel paint. The gloss of a paint surface is affected by coarse particles in a pigment used. The quality of a pigment, in this respect, is best observed by actually preparing the paint under standardized conditions and optically examining the surface. The products of the example and the control were incorporated in a gloss sensitive, alkyd-amino resin vehicle (Ford M30–J automotive baking enamel) and thinned with volatile solvent to spraying consistency and sprayed onto panels. After drying and baking, the panels were compared in a standardized gloss meter. In the gloss meter a focused light beam was directed onto the enamel surface at a 20 degree incident angle. A photoelectric meter was placed to intercept the reflected beam and measure its intensity. Higher readings on the meter indicated the better gloss since surface imperfections cause diffused reflection which does not reach the photometer. The gloss meter was standardized with respect to the intensity of incident light and the portion of reflected light reaching the photocell, against a glass plate corresponding to a value on an arbitrary gloss scale. The experimental samples and controls may thus be compared and given a significant numerical relative gloss rating.

Sometimes the dry grinding of titanium dioxide pigment, for example in impact and ring-roller mills, will cause degradation of the carbon black undertone. As shown in this example the method of this invention gives an improved level of carbon black undertone.

Example 2

Micronizers of the same size and design as used in Example 1 were employed to process TiO_2 pigment manufactured by the oxidation of vaporous titanium tetrachloride. The feed pigment was dry and contained 0.15% of grit retained on a 325 mesh screen after wet screening. Steam was supplied to the nozzzles of the improved mill at 300 p.s.i. and 50° F. of superheat; steam to the conventional mill was at 170 p.s.i. to maintain the same rate. The calculated steam velocity leaving the nozzles in the improved mill was 1.96 Mach., total nozzles steam flow for each mill 3500 #/hr., and the pigment injector steam was used at a rate of 1800 #/hr. for each mill. Each mill was fed under these conditions

at pigment rates as shown below, with results as indicated.

Mill	Supersonic			Conventional		
#TiO_2/hr.	1,500	1,900	2,500	1,500	1,900	2,500
Gloss	80	78	77	77	75	74
Color	24:4*y*			24:4*y*		
Grit	Too low to measure			Very low		

I claim:

1. In a process for comminuting grit in a pigment the steps comprising (1) injecting a gaseous fluid tangentially and at supersonic velocity into the periphery of an enclosed, circular space having only an axial outlet, whereby to establish in said circular space a disc-shaped vortex of said gaseous fluid inwardly spiraling to said outlet, (2) injecting grit-containing pigment into said vortex at a locus remote from said outlet, whereby the grit is completely comminuted in the vortex, and (3) discharging the gaseous fluid, pigment, and comminuted grit through the outlet while maintaining the pressure therein at 2 to 30 p.s.i.a.

2. A process of claim 1 wherein the gaseous fluid is air.

3. A process of claim 1 wherein the gaseous fluid is steam.

4. In a process for comminuting grit in a titanium dioxide pigment the steps comprising (1) injecting superheated steam tangentially, at supersonic velocity, and at a multiplicity of substantially equally spaced points, into the periphery of an enclosed, circular space having only an axial outlet, whereby to establish therein a disc-shaped vortex of steam inwardly spiraling to said outlet, (2) injecting grit-containing titanium dioxide pigment into said vortex at a locus remote from said outlet, and (3) discharging the steam, pigment, and comminuted grit through the said axial outlet while maintaining the pressure therein at 2 to 30 p.s.i.a.

5. A process of claim 4 wherein the amount of steam injected into the periphery at supersonic velocity is at least 15 pounds per hour per square inch of vortex rim area and the weight ratio of injected steam to pigment is from 0.5:1 to 3:1.

6. In an improved apparatus for comminuting pulverulent solids, in combination, a circular, disc-shaped chamber having a peripheral wall through which said chamber is in closed communication with a source of pressurized gaseous fluid through a multiplicity of converging-diverging nozzles, each nozzle having inlet bore, throat bore, and exit bore portions, all of circular cross section and having a common axis, the inlet bore converging through a neck to a cylindrical throat having a diameter about one-third that of the inlet bore, said throat opening into a tapered outlet bore of diameter increasing linearly in the direction of a discharge outlet in said exit portion, the taper angle between the wall and axis of the outlet bore being about 2 to 14°, said exit portion having a truncated exit face, the angle between the truncating plane forming the exit face and a plane perpendicular to the axis of the outlet bore being about from 19 to 42°, and there being in the truncated face an inset face around the discharge opening of the outlet bore, perpendicular to the axis thereof and extending out from said discharge opening a distance of from ½ to 3 radii of said opening, the opening defined by said inset face tapering outwardly from the periphery of said face to the truncated face at an angle of at least about 6° from the axis of the outlet bore, said nozzles being mounted in said peripheral wall at substantially equally spaced points therein, with the truncated face of each nozzle substantially flush with said wall and the axis of the nozzle bore being tangent to a circle concentric with said chamber, of a radius from ⅓ to ⅔ the radius of said chamber, the chamber having an axial discharge port concentric with its periphery,

means communicating with said chamber for feeding pulverulent solids into it between its periphery and the discharge port, and means communicating with the discharge port for separating effluent solids from gaseous fluids.

7. The apparatus of claim 6 wherein the disc-shaped chamber is axially connected through a duct to means for lowering the pressure in said chamber at the discharge port.

8. The apparatus of claim 6 wherein the taper angle between the wall and axis of the outlet bore is about 6°, the angle between the truncating plane forming the exit face and a plane perpendicular to the axis of the outlet bore is about 30°, and the inset face around the discharge opening of the outlet bore, perpendicular to the axis thereof, has a diameter about twice the diameter of said discharge opening, and the opening defined by said inset face tapers outwardly from the periphery of said face to the truncated face at an angle of about 15° from the axis of the outlet bore.

9. A converging-diverging nozzle adapted for injecting a gaseous fluid at supersonic velocity into a vortex, the nozzle comprising inlet bore, throat bore, and exit bore portions, all of circular cross section and having a common axis, the inlet bore converging through a neck to a cylindrical throat having a diameter about one-third that of the inlet bore, said throat opening into a tapered outlet bore of diameter increasing linearly in the direction of a discharge outlet in said exit portion, the taper angle between the wall and axis of the outlet bore being about 2 to 14°, said exit portion having a truncated exit face, the angle between the truncating plane forming the exit face and a plane perpendicular to the axis of the outlet bore being about from 19 to 42°, and there being in the truncated face an inset face around the discharge opening of the outlet bore, perpendicular to the axis thereof and extending out from said discharge opening a distance of from ½ to 3 radii of said opening, the opening defined by said inset face tapering outwardly from the periphery of said face to the truncated face at an angle of at least about 6° from the axis of the outlet bore.

10. A converging-diverging nozzle adapted for injecting a gaseous fluid at supersonic velocity into a vortex, the nozzle comprising inlet bore, throat bore, and exit bore portions, all of circular cross section and having a common axis, the inlet bore converging through a neck to a cylindrical throat having a diameter about one-third that of the inlet bore, said throat opening into a tapered outlet bore of diameter increasing linearly in the direction of a discharge outlet in said exit portion, the taper angle between the wall and axis of the outlet bore being about 6°, said exit portion having a truncated exit face, the angle between the truncating plane forming the exit face and a plane perpendicular to the axis of the outlet bore being about 30°, and there being in the truncated face a circular inset face around the discharge opening of the outlet bore, perpendicular to the axis thereof and about twice the diameter of said discharge opening, the opening defined by said inset face tapering outwardly from the periphery of said face to the truncated face at an angle of about 15° from the axis of the outlet bore.

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